



10/506458
Rec'd PCT/PTO 01 SEP 2004
PCT/AU03/00250

REC'D 19 MAR 2003

WIPO

PCT

PRIORITY DOCUMENT
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH
RULE 17.1(a) OR (b)

**Patent Office
Canberra**

I, SMILJA DRAGOSAVLJEVIC, TEAM LEADER EXAMINATION
SUPPORT AND SALES hereby certify that annexed is a true copy of the
Provisional specification in connection with Application No. PS 0855 for a
patent by THE UNIVERSITY OF NEWCASTLE RESEARCH ASSOCIATES
LIMITED as filed on 01 March 2002.

WITNESS my hand this
Tenth day of March 2003

S. Dragosavljevic

SMILJA DRAGOSAVLJEVIC
TEAM LEADER EXAMINATION
SUPPORT AND SALES



BEST AVAILABLE COPY

AUSTRALIA

PATENTS ACT 1990

PROVISIONAL SPECIFICATION

FOR THE INVENTION ENTITLED:-

"SYNTACTIC FOAM"

The invention is described in the following statement:-

The present invention relates to a method of manufacturing low density syntactic foam.

BACKGROUND OF THE INVENTION

Syntactic foam is made up of a mix of pre-formed micro-spheres and resin.

- 5 Syntactic foams have been used in areas where low densities are required with high strength as in undersea/marine equipment for deep-ocean current metering, anti-submarine warfare and sandwich composites.

The process of manufacturing syntactic foams is different from that of conventional foams. In one known manufacturing process, the consolidation method for
10 binder and micro-spheres includes the coating of micro-spheres, prior to which are the steps of vacuum filtering and rinsing. Other manufacturing processes make use of inorganic binder solution and firing, dry resin powder for sintering, and liquid resin as binder for in-situ reaction injection moulding.

- Another known manufacturing process for syntactic foam has been developed.
15 using a compaction method which includes a mixture of liquid resin/micro-spheres achieving a resin volume fraction of 0.09 and a density of 0.6g/cc.

A slip casting method employing porous plaster moulds to drain excessive liquid binder has also been developed. An advantage of this method is its potentially suitability for the manufacture of thick items while the disadvantages could be the limited service
20 life of the plaster mould and poor surface finish.

The present invention is a new manufacturing method suitable but not limited to the manufacture of syntactic foam, in which the natural characteristics of the micro-spheres are used to obtain reduced densities while maintaining desired strength. The syntactic foam density can be lowered down to 0.08 which is very close to its micro-
25 sphere bulk density of 0.72.

Any discussion of the prior art throughout the specification should in no way be considered as an admission that such prior art is widely known or forms part of common general knowledge in the field.

DISCLOSURE OF THE INVENTION

5 According to the present invention there is provided a method of manufacturing syntactic foam including the steps of:
providing a pre-determined ratio of constituent materials including micro-spheres and a liquid phase binder;

 mixing the constituent materials in a container;
10 casting said constituents in a mould;
 allowing the volume of the liquid phase binder between the micro-spheres to be reduced by the natural characteristic of the buoyant force of each micro-sphere
 squeezing out excess liquid phase binder from between the micro-spheres, and
 draining the excess liquid phase binder from the mould.

15 Preferably, mixing takes place by agitation.

 Preferably, agitation is achieved by shaking the container.

 Preferably, the container has a closed top.

 Preferably, the mould has an inner surface of a desired physical shape to achieve moulding of a predetermined product.

20 Preferably, the excess liquid phase binder is drained from the bottom of the mould.

 Preferably, said liquid phase binder is any combination of acetone, epoxy and a hardener.

 Preferably, micro-spheres are hollow spheres.

25 BRIEF DESCRIPTION OF THE DRAWINGS

A preferred embodiment of the invention will now be described, by way of example only, with reference to the accompanying drawings in which:

Fig 1a) is a schematic of the dispersed micro-spheres and liquid phase binder immediately after being cast into the mould.

5 Fig 1b) is a similar view to Fig 1a) after a period of time such that the micro-spheres are partially packed due to the buoyancy effect.

Fig 1c) is a similar view to Fig 1b) after a further period of time such that the micro-spheres are fully packed.

Fig 1d) is a similar view to Fig 1c) after an additional further period showing the
10 fully packed layer of micro-spheres gravitated down the mould after the liquid phase binder is drained through an orifice at the bottom of said mould.

PREFERRED EMBODIMENTS OF THE INVENTION

Although the invention is described with reference to specific examples, it will be appreciated by those skilled in the art that the invention may be embodied in many
15 other forms.

In the laboratory, a method of manufacturing syntactic foam typically includes the steps of, mixing epoxy hardener acetone by first placing a clean, empty mixing container on an electronic scale, and then injecting a pre-mixed epoxy hardener into the container using a plastic syringe until the required mass is reached. Acetone is then
20 added and the container is then sealed and shaken vigorously for 2 minutes. The container is then opened and the micro-spheres then added through a glass funnel. The container is then sealed and shaken again vigorously for a further 5 minutes to disperse the micro-spheres. For subsequent casting, the container is then kept shaken to maintain a constant mixture ration whilst the mixture is being poured through a tube into a mould.
25 Referring to figure 1(a), a mixture of micro-spheres 1 and liquid phase binder 2 are

placed into a mould 3 such that the micro-spheres 1 are dispersed evenly within the liquid phase binder 2 immediately after being placed into the mould. The micro-spheres 1 which are in suspension with the liquid phase binder 2 begin by the natural buoyancy effect to move to an upper surface of the mould as shown in figure 1(b). After a further period of time the micro-spheres 1 begin to self pack into a layer 4 as shown in figure 1(c) by the natural effect of the buoyancy force exerted on each micro-sphere immersed in the liquid phase binder 2. After a further period of time when the micro-spheres are fully packed into a layer 4 as indicated in figures 1(c) the liquid phase binder settles to the bottom of the mould. After a further period of time the liquid phase binder 2 is then drained from the bottom of the mould, preferably through an opening that is situated at the bottom of the mould 3 as shown in figure 1(d), the self packed layer of micro-spheres 4 slowly gravitate to the bottom of the mould and take up a predetermined shape defined by the mould 3 with the self packed micro-spheres 1 forming a layer 5 at the bottom of the mould. After 30 minutes, the foam in the mould is sufficiently dry so it is demoulded.

Densities of manufactured syntactic foam with various amounts of constituent materials are listed in figure 2 Table 1. From Table 1, we see the measured bulk density. Different mixing ratios were employed by varying acetone content but keeping the mass ratio of micro-spheres to (epoxy+hardener) constant as 1 to 2 as seen in the second column of figure 2 Table 1. As can be seen from Table 1, the foam density decreases as the acetone content increases, and very closely approaches the bulk density of the micro-spheres which are the lower limit of the achievable foam density. In general, since the density of liquid phase is much higher than that of micro-spheres, a low foam density can be achieved by reducing the amount of liquid phase or increasing packing

density of micro-spheres. The higher the buoyant force, the higher the packing is expected unless there are other factors contributing to the packing.

While the buoyant force may be a main driving force in forming the current syntactic foam, factors affecting the buoyant force may include viscosity and densities of
5 constituent materials. The net buoyant force may be reduced in the presence of a viscous drag such that

$$\text{NBF (net buoyant force)} = \text{BF (buoyant force)} - \text{viscous drag.}$$

The viscous drag increases with increasing viscosity. However, as the acetone
content increases, the viscous drag decreases due to the decrease in viscosity of liquid
10 mixture as shown. Concurrently, the BF also decreases because of the decrease in density of liquid phase. Thus, the improvement in lowering the foam density by the addition of acetone appears to be due to more decrease in the viscous drag than that in the BF, which results in increased NBF. Thus a new manufacturing method using a BF
technique has been developed for syntactic foam. It has been demonstrated that the
15 syntactic foam density can be lowered down to 0.08 which is very close to its micro-sphere bulk density of 0.72.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method of manufacturing syntactic foam including the steps of:
providing a pre-determined ratio of constituent materials including micro-spheres and a liquid phase binder;
5 mixing the pre-determined constituent materials in a container;
casting said constituents in a mould;
allowing the volume of the liquid phase binder between the micro-spheres to be reduced by the natural characteristic of the buoyant force of each micro-sphere squeezing out excess liquid phase binder from between the micro-spheres, and
10 draining the excess liquid phase binder from the mould.
2. The method according to claim 1 wherein mixing of the pre-calculated constituent materials takes place by agitation.
3. The method according to claim 1 or claim 2, wherein agitation is achieved by shaking the container.
- 15 4. The method according to any one of the preceding claims, wherein said container has a closed top.
5. The method according to any one of the preceding claims, wherein said mixture is placed in a mould having an inner surface of the desired physical shape to achieve moulding of a pre-determined product.
- 20 6. The method according to any one of the preceding claims, wherein the excess liquid phase binder is drained from the bottom of the mould.
7. The method according to any one of the preceding claims, wherein said liquid phase binder is any combination of acetone, epoxy and a hardener.
8. The method according to any one of the preceding claims, wherein said micro-
25 spheres are hollow spheres.

9. The method substantially as herein described with reference to the accompanying drawings.

Dated this 1st Day of March 2002

THE UNIVERSITY OF NEWCASTLE RESEARCH ASSOCIATES LIMITED

5

Attorney: JOHN D. FORSTER
Fellow Institute of Patent and Trade Mark Attorneys of Australia
of BALDWIN SHELSTON WATERS

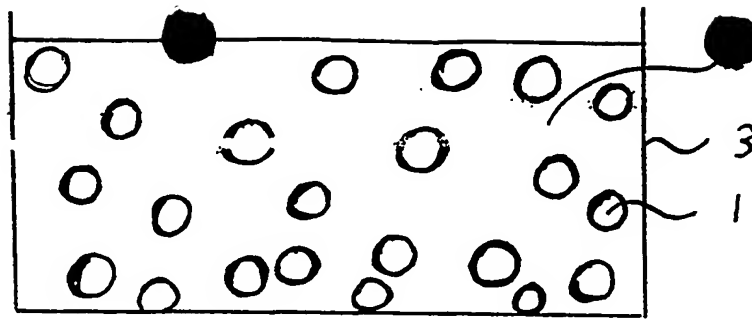


FIG 1 a)

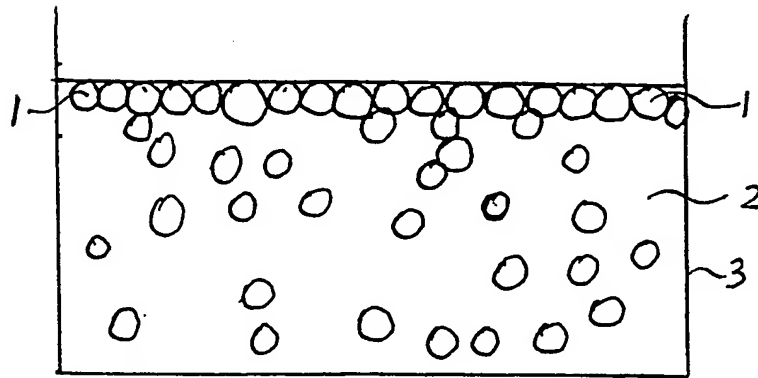


FIG 1 b)

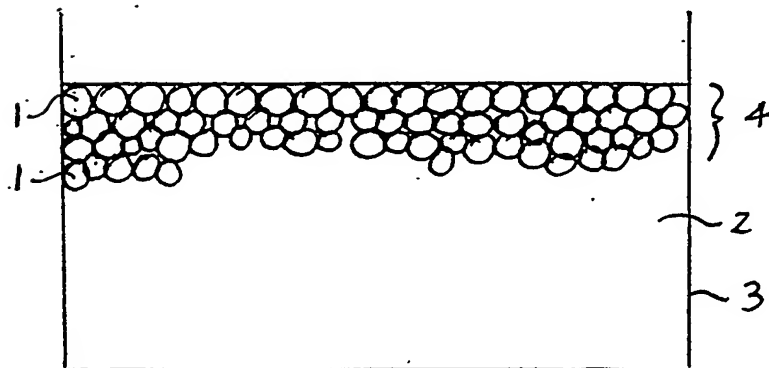


FIG 1 c)

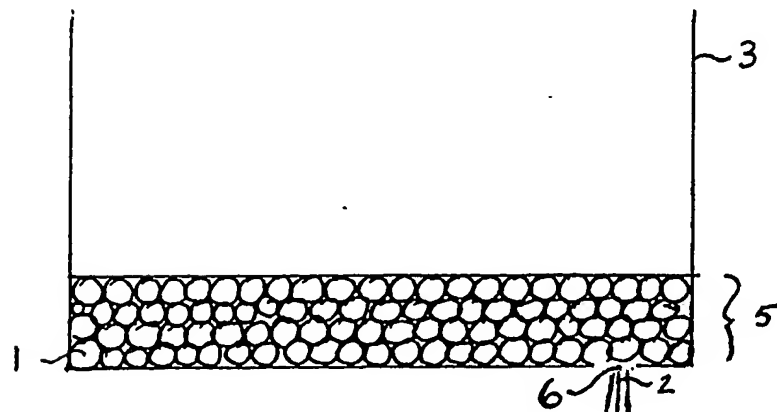


FIG 1 d)

FIGURE 2

TABLE 1

Mould No.	Mass ratio (acetone/microspheres/ (epoxy+hardener))	Volume fraction ratio (acetone/microspheres/ (epoxy+hardener))	Foam bulk density (g/cc)
1	6/1/2	0.44/0.45/0.106	0.250
2	8/1/2	0.51/0.40/0.092	0.137
3	10/1/2	0.57/0.35/0.082	0.112
4	12/1/2	0.61/0.31/0.074	0.123
5	14/1/2	0.65/0.29/0.067	0.106
6	18/1/2	0.70/0.24/0.056	0.096
7	30/1/2	0.80/0.16/0.038	0.082
8	40/1/2	0.84/0.13/0.030	0.080

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☐ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.